

§ 178.3900

21 CFR Ch. I (4–1–05 Edition)

§ 178.3900 Sodium pentachlorophenate.

Sodium pentachlorophenate may be safely used as a preservative for ammonium alginate employed as a processing aid in the manufacture of polyvinyl chloride emulsion polymers intended for use as articles or components of articles that contact food at temperatures not to exceed room temperature. The quantity of sodium pentachlorophenate used shall not exceed 0.5 percent by weight of ammonium alginate solids.

§ 178.3910 Surface lubricants used in the manufacture of metallic articles.

The substances listed in this section may be safely used in surface lubri-

cants employed in the manufacture of metallic articles that contact food, subject to the provisions of this section.

(a) The following substances may be used in surface lubricants used in the rolling of metallic foil or sheet stock provided that total residual lubricant remaining on the metallic article in the form in which it contacts food does not exceed 0.015 milligram per square inch of metallic food-contact surface:

(1) Substances identified in paragraphs (b)(1) and (2) of this section.

(2) Substances identified in this paragraph.

List of substances	Limitations
<p>α-Butyl-Ω-hydroxypoly (oxyethylene)-poly (oxypropylene) (CAS Reg. No. 9038–95–3) produced by random condensation of a 1:1 mixture by weight of ethylene oxide and propylene oxide with butanol and having a minimum molecular weight of 1,000.</p> <p>α-Butyl-Ω-hydroxypoly(oxypropylene) (CAS Reg. No. 9003–13–8) having a minimum molecular weight of 1000.</p> <p>α-Lauroyl-Ω-hydroxypoly(oxyethylene) (CAS Reg. No. 9004–81–3) having a minimum molecular weight of 200.</p> <p>Acetate esters derived from synthetic straight chain alcohols (complying with § 172.864 of this chapter) that have even numbers of carbon atoms in the range C₈–C₁₈.</p> <p>α-Alkyl-ω-hydroxypoly(oxyethylene) produced by the condensation of 1 mole of C₁₂–C₁₅ straight chain primary alcohols with an average of 3 moles of ethylene oxide (CAS Reg. No. 68002–97–1).</p> <p>Benzotriazole (CAS Reg. No. 95–14–7)</p> <p>Bis(hydrogenated tallow alkyl)amine (CAS Reg. No. 61789–79–5).</p> <p>Bis(hydrogenated tallow alkyl)aminoethanol (CAS Reg. No. 116438–56–3).</p> <p><i>N,N</i>-Bis(2-hydroxyethyl)butylamine (CAS Reg. No. 102–79–4).</p> <p><i>Tert</i>-Butyl alcohol.</p> <p>Di(2-ethylhexyl)phthalate.</p> <p>Diethyl phthalate.</p> <p>Diethylene glycol monobutylether (CAS Reg. No. 112–34–5).</p> <p>Dimers, trimers, and/or their partial methyl esters; such dimers and trimers are of unsaturated C₁₈ fatty acids derived from animal and vegetable fats and oils and/or tall oil, and such partial methyl esters meet the following specifications: Saponification value 180–200, acid value 70–130, and maximum iodine value 120.</p> <p>Di-<i>n</i>-octyl sebacate.</p> <p>Ethylenediaminetetraacetic acid, sodium salts.</p> <p>Isopropyl alcohol.</p> <p>Isopropyl laurate (CAS Reg. No. 10233–13–3)</p> <p>Isopropyl oleate.</p> <p>Isotridecyl alcohol, ethoxylated (CAS Reg. No. 9043–30–5).</p> <p>Methyl esters of coconut oil fatty acids.</p> <p>Methyl esters of fatty acids (C₁₆–C₁₈) derived from animal and vegetable fats and oils.</p> <p>Polybutene, hydrogenated: complying with the identity prescribed under § 178.3740(b).</p> <p>Polyethylene glycol (400) monostearate.</p> <p>Polyisobutylene (minimum molecular weight 300).</p>	<p>Not to be used in combination with sodium nitrite.</p> <p>For use only at a level not to exceed 10 percent by weight of finished lubricant formulation.</p> <p>For use at a level not to exceed 10 percent by weight of the finished lubricant formulation.</p>

Food and Drug Administration, HHS

§ 178.3910

List of substances	Limitations
<p>Polyoxyethylated (5 moles) tallow amine (CAS Reg. No. 61791-26-2).</p> <p>Polyvinyl alcohol.</p> <p>Sodium nitrite</p>	<p>For use only as a rust inhibitor in lubricant formulations provided the total residual sodium nitrite on the metallic article in the form in which it contacts food does not exceed 0.007 milligram per square inch of metallic food-contact surface.</p>
<p>Sodium petroleum sulfonate, MW 440-450 (CAS Reg. No. 68608-26-4) derived from naphthenic oil having a Saybolt viscosity range of 500-600 Saybolt Universal Seconds (SUS at 37-8 °C (100 °F) as determined by ASTM method D88-81, "Standard Test Method for Saybolt Viscosity," which is incorporated by reference. Copies are available from the American Society for Testing Materials, 1961 Race St., Philadelphia, PA 19103, or available for inspection at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202-741-6030, or go to: http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.</p> <p>Synthetic alcohol mixture of straight- and branched-chain alcohols that have even numbers of carbon atoms in the range C₄-C₁₈ and that are prepared from ethylene, aluminum, and hydrogen such that the finished synthetic alcohol mixture contains not less than 75 pct of straight-chain primary alcohols and contains not less than 85 pct total C₁₀ and C₁₂ alcohols.</p> <p>Synthetic primary alcohol mixture of straight- and branched-chain alcohols that contain at least 99 pct primary alcohols consisting of the following: not less than 70 pct normal alcohols; not less than 96.5 pct C₁₂-C₁₅ alcohols; and not more than 2.5 pct alpha, omega C₁₃-C₁₆ diols. The alcohols are prepared from linear olefins from a purified kerosene fraction, carbon monoxide and hydrogen using a modified oxo process, such that the finished primary alcohol mixture meets the following specifications: Molecular weight, 207±4; hydroxyl number, 266-276.</p> <p>Synthetic primary alcohol mixture of straight- and branched-chain alcohols that contain at least 99 pct primary alcohols consisting of the following: not less than 70 percent normal alcohols; not less than 93 pct C₁₂-C₁₃ alcohols; not more than 5 pct C₁₄-C₁₅ alcohols; and not more than 2.5 pct alpha, omega, C₁₃-C₁₆ diols. The alcohols are prepared from linear olefins from a purified kerosene fraction, carbon monoxide and hydrogen using a modified oxo process, such that the finished primary alcohol mixture meets the following specifications:</p> <p>Molecular weight 194±5; hydroxyl number, 283-296.</p> <p>Tallow, sulfonated.</p> <p>Triethanolamine.</p>	<p>For use at a level not to exceed 8 pct by weight of the finished lubricant formulation.</p> <p>For use only at a level not to exceed 8 pct by weight of the finished lubricant formulation.</p>

(3) Mineral oil conforming to the identity prescribed in §178.3620(c).

(4) Light petroleum hydrocarbons identified in paragraph (a)(4) (i) of this section: *Provided*, That the total residual lubricant on the metallic article in the form in which it contacts food meets the ultraviolet absorbance limits prescribed in paragraph (a) (4) (ii) of this section as determined by the analytical method described in paragraph (a) (4) (iii) of this section.

(i) Light petroleum hydrocarbons are derived by distillation from virgin petroleum stocks or are synthesized from petroleum gases. They are chiefly par-

affinic, isoparaffinic, naphthenic, or aromatic in nature, and meet the following specifications:

(a) Initial boiling point is 24 °C minimum and final boiling point is 288 °C maximum, as determined by ASTM method D86-82, "Standard Method for Distillation of Petroleum Products," which is incorporated by reference. Copies may be obtained from the American Society for Testing Materials, 1916 Race St., Philadelphia, PA 19103, or may be examined at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call

§ 178.3910

21 CFR Ch. I (4-1-05 Edition)

202-741-6030, or go to: http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html.

(b) Nonvolatile residue is 0.005 gram per 100 milliliters, maximum, as determined by ASTM method D381-80, "Standard Test Method for Existent Gum in Fuels by Jet Evaporation," when the final boiling point is 121 °C or above and by ASTM method D1353-78, "Standard Test Method for Nonvolatile Matter in Volatile Solvents for Use in Paint, Varnish, Lacquer, and Related Products," when the final boiling point is below 121 °C. These ASTM methods are incorporated by reference. The availability of these incorporations by reference is given in paragraph (a)(4)(i)(a) of this section.

(c) Saybolt color 20 minimum as determined by ASTM method D156-82, "Standard Test Method for Saybolt Color of Petroleum Products (Saybolt Chromometer Method)," which is incorporated by reference. The availability of this incorporation by reference is given in paragraph (a)(4)(i)(a) of this section.

(d) Aromatic component content shall not exceed 32 percent.

(e) Conforms with ultraviolet absorbance limits prescribed in §178.3620(c) as determined by the analytical method described therein.

(ii) Ultraviolet absorbance limits on residual lubricants are as follows:

Wavelength (mμ)	Maximum absorbance per 5 centimeters optical pathlength
280-289	0.7
290-2996
300-3594
360-40009

(iii) The analytical method for determining ultraviolet absorbance limits on residual lubricants is as follows:

GENERAL INSTRUCTIONS

Because of the sensitivity of the test, the possibility of errors arising from contamination is great. It is of the greatest importance that all glassware be scrupulously cleaned to remove all organic matter such as oil, grease, detergent, residues, etc. Examine all glassware including stoppers and stopcocks, under ultraviolet light to detect any residual

fluorescent contamination. As a precautionary measure it is recommended practice to rinse all glassware with purified isooctane immediately before use. No grease is to be used on stopcocks or joints. Great care to avoid contamination of oil samples in handling and to assure absence of any extraneous material arising from inadequate packaging is essential. Because some of the polynuclear hydrocarbons sought in this test are very susceptible to photo-oxidation, the entire procedure is to be carried out under subdued light.

APPARATUS

Separatory funnels. 250-milliliter, 500-milliliter, 1,000-milliliter, and preferably 2,000-milliliter capacity, equipped with tetrafluoroethylene polymer stopcocks.

Evaporation flask (optional). 250-milliliter or 500-milliliter capacity all-glass flask equipped with standard-taper stopper having inlet and outlet tubes to permit passage of nitrogen across the surface of contained liquid to be evaporated.

Spectrophotometric cells. Fused quartz cells, optical path length in the range of 5,000 centimeters ± 0.005 centimeter; also for checking spectrophotometer performance only, optical path length in the range 1.000 centimeter ± 0.005 centimeter. With distilled water in the cells, determine any absorbance differences.

Spectrophotometer. Special range 250 millicrons-400 millimicrons with spectral slit width of 2 millimicrons or less; under instrument operating conditions for these absorbance measurements, the spectrophotometer shall also meet the following performance requirements:

Absorbance repeatability, ± 0.01 at 0.4 absorbance.

Absorbance accuracy,¹ ± 0.05 at 0.4 absorbance.

Wavelength repeatability, ± 0.2 millimicron.

Wavelength accuracy, ± 1.0 millimicron.

¹As determined by procedure using potassium chromate for reference standard and described in National Bureau of Standards Circular 484, Spectrometry, U.S. Department of Commerce (1949), which is incorporated by reference. Copies are available from the Center for Food Safety and Applied Nutrition (HFS-200), Food and Drug Administration, 5100 Paint Branch Pkwy., College Park, MD 20740, or available for inspection at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202-741-6030, or go to: http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html. The accuracy is to be determined by comparison with the standard values at 210, 345, and 400 millimicrons.

Soxhlet apparatus. 60-millimeter diameter body tubes fitted with condenser and 500-milliliter round-bottom boiling flask. A supply of paper thimbles to fit is required.

Nitrogen cylinder. Water-pumped or equivalent purity nitrogen in cylinder equipped with regulator and valve to control flow at 5 p.s.i.g.

REAGENTS AND MATERIALS

Organic solvents. All solvents used throughout the procedure shall meet the specifications and tests described in this specification. The isooctane (2,2,4-trimethylpentane) shall pass the following test:

Place 180 milliliters of solvent in a 250-milliliter Erlenmeyer flask, add 1 milliliter of purified *n*-hexadecane and evaporate on the steam bath under a stream of nitrogen (a loose aluminum foil jacket around the flask will speed evaporation). Discontinue evaporation when not over 1 milliliter of residue remains.

Alternatively, the evaporation time can be reduced by using the optional evaporation flask. In this case the solvent and *n*-hexadecane are placed in the flask on the steam bath, the tube assembly is inserted, and a stream of nitrogen is fed through the inlet tube while the outlet tube is connected to a solvent trap and vacuum line in such a way as to prevent any flow-back of condensate into the flask.

Dissolve the 1 milliliter of hexadecane residue in isooctane and make to 25 milliliters volume. Determine the absorbance in the 5-centimeter path length cells compared to isooctane as reference. The absorbance of the solution of the solvent residue shall not exceed 0.01 per centimeter path length between 280 and 400 mμ. Purify, if necessary, by passage through a column of activated silica gel (Grade 12, Davison Chemical Co., Baltimore, Maryland, or equivalent) about 90 centimeters in length and 5 centimeters to 8 centimeters in diameter.

n-Hexadecane, 99-percent olefin-free. Dilute 1.0 milliliter of *n*-hexadecane to 25 milliliters with isooctane and determine the absorbance in a 5-centimeter cell compared to isooctane as reference point between 280 mμ-400 mμ. The absorbance per centimeter path length shall not exceed 0.00 in this range. Purify, if necessary, by percolation through activated silica gel or by distillation.

Dimethyl sulfoxide. Spectrophotometric grade (Crown Zellerbach Corp., Camas, Washington, or equivalent). Absorbance (1-centimeter cell, distilled water reference, sample completely saturated with nitrogen).

Wavelength	Absorbance (maximum)
261.5	1.00
27020
27509

Wavelength	Absorbance (maximum)
28006
300015

There shall be no irregularities in the absorbance curve within these wavelengths.

Phosphoric acid. 85 percent A.C.S. reagent grade.

Sodium sulfate, anhydrous, A.C.S. reagent grade, preferably in granular form. For each bottle of sodium sulfate reagent used, establish as follows the necessary sodium sulfate prewash to provide such filters required in the method: Place approximately 35 grams of anhydrous sodium sulfate in a 30-milliliter coarse, fritted-glass funnel or in a 65-milliliter filter funnel with glass wool plug; wash with successive 15-milliliter portions of the indicated solvent until a 15-milliliter portion of the wash shows 0.00 absorbance per centimeter path length between 280 mμ and 400 mμ when tested as prescribed under "Organic solvents." Usually three portions of wash solvent are sufficient.

Before proceeding with analysis of a sample, determine the absorbance in a 5-centimeter path cell between 250 millimicrons and 400 millimicrons for the reagent blank by carrying out the procedure, without a metal sample. The absorbance per centimeter path length should not exceed 0.02 in the wavelength range from 280 mμ to 400 mμ.

Place 300 milliliters of dimethyl sulfoxide in a 1-liter separatory funnel and add 75 milliliters of phosphoric acid. Mix the contents of the funnel and allow to stand for 10 minutes. (The reaction between the sulfoxide and the acid is exothermic. Release pressure after mixing, then keep funnel stoppered.) Add 150 milliliters of isooctane and shake to pre-equilibrate the solvents. Draw off the individual layers and store in glass-stoppered flasks.

PROCEDURE

Sample. Select metal foil or sheet stock for the test which has not been previously contaminated by careless handling or exposure to atmospheric dust and fumes. A commercial coil in the form supplied for spindle mounting in a packaging line or wrapping machine is most suitable. Strip off the outside turn of metal and discard. Carefully avoid contamination or damage from handling the metal (wear gloves). Remove a 16-18-foot length from the coil and place it on a flat surface protected by a length of new kraft paper. Cut four 15-foot strips from the sample, each 3 inches wide (avoid tearing the edges of the strips). Using a piece of suitable glass rod, roll the strips of metal into loose coils and insert each into a Soxhlet thimble. Each turn of coil should be visibly separated from the adjacent turn.

Extraction. Fill each of the four Soxhlet tubes with purified isooctane (see under heading “Reagents and Materials,” above) until siphon action occurs and then refill the tube body. Supply heat to the boiling flask and allow extraction to continue for at least 8 hours or until repeated weighings of the dried and cooled coil show no further weight loss.

Combine the isooctane extracts from the four Soxhlet units in a suitable beaker, rinsing each tube and flask into the beaker with fresh purified solvent. Evaporate the solvent under an atmosphere of inert gas (nitrogen) to residual volume of 50–60 milliliters and transfer this solution to a 500-milliliter separatory funnel containing 100 milliliters of pre-equilibrated sulfoxide-phosphoric acid mixture. Complete the transfer of the sample with small portions of pre-equilibrated isooctane to give a total volume of the residue and solvent of 75 milliliters. Shake the funnel vigorously for 2 minutes. Set up three 250-milliliter separatory funnels with each containing 30 milliliters of pre-equilibrated isooctane. After separation of liquid phases, carefully draw off lower layer into the first 250-milliliter separatory funnel and wash in tandem with the 30-milliliter portion of isooctane contained in the 250-milliliter separatory funnels. Shaking time for each wash is 1 minute. Repeat the extraction operation with two additional portions of the sulfoxide-acid mixture and wash each extractive in tandem through the same three portions of isooctane.

Collect the successive extractives (300 milliliters total) in a separatory funnel (preferably 2-liter) containing 480 milliliters of distilled water; mix, and allow to cool for a few minutes after the last extractive has been added. Add 80 milliliters of isooctane to the solution and extract by shaking the funnel vigorously for 2 minutes. Draw off the lower aqueous layer into a second separatory funnel (preferably 2-liter) and repeat the extraction with 80 milliliter of isooctane. Draw off and discard the aqueous layer. Wash each of the 80 milliliter extractives three times with 100-milliliter portions distilled water. Shaking time for each wash is 1 minute. Discard the aqueous layers. Filter the first ex-

tractive through anhydrous sodium sulfate pre-washed with isooctane (see sodium sulfate under “Reagents and Materials” for preparation of filter) into a 250-milliliter Erlenmeyer flask (or optionally into the evaporation flask). Wash the first separatory funnel with the second 80-milliliter isooctane extractive and pass through the sodium sulfate. Then wash the second and first separatory funnels successively with a 20-milliliter portion of isooctane and pass the solvent through the sodium sulfate into the flask. Add 1 milliliter of *n*-hexadecane and evaporate the isooctane on the steam bath under nitrogen. Discontinue evaporation when not over 1 milliliter of residue remains. To the residue, add a 10-milliliter portion of isooctane, reevaporate to 1 milliliter of hexadecane, and repeat this operation once.

Quantitatively transfer the residue with isooctane to a 25-milliliter volumetric flask, make to volume, and mix. Determine the absorbance of the solution in 5-centimeter pathlength cells compared to isooctane as reference between 280mμ–400mμ (take care to lose none of the solution in filling the sample cell). Correct the absorbance values for any absorbance derived from reagents as determined by carrying out the procedure without a metal sample. If the corrected absorbance does not exceed the limits prescribed in this paragraph, the residue meets the ultraviolet absorbance specifications.

(b) The following substances may be used in surface lubricants used to facilitate the drawing, stamping, or forming of metallic articles from rolled foil or sheet stock by further processing provided that the total residual lubricant remaining on the metallic article in the form in which it contacts food does not exceed 0.2 milligram per square inch of food-contact surface:

(1) Antioxidants used in compliance with regulations in parts 170 through 189 of this chapter.

(2) Substances identified in this subparagraph.

List of substances	Limitations
Acetyl tributyl citrate. Acetyl triethyl citrate. Butyl stearate. Castor oil. Dibutyl sebacate. Di(2-ethylhexyl) azelate. Di(2-ethylhexyl) sebacate. Diisodecyl phthalate. Dimethylpolysiloxane	Conforming to the identity prescribed in § 181.28 of this chapter.
Dipropylene glycol. Epoxidized soybean oil	Conforming to the identity prescribed in § 181.27 of this chapter.

List of substances	Limitations
Fatty acids derived from animal and vegetable fats and oils, and salts of such acids, single or mixed, as follows: Aluminum Magnesium Potassium Sodium Zinc	
Fatty alcohols, straight-chain with even number carbon atoms (C ₁₀ or greater).	
Isobutyl stearate.	
Lanolin.	
Linoleic acid amide.	
Mineral oil	Conforming to the identity prescribed in § 178.3620 (a) or (b).
Mono-, di-, and tristearyl citrate.	
Oleic acid amide.	
Palmitic acid amide.	
Petrolatum	Conforming to the identity prescribed in § 178.3700.
Phosphoric acid, mono- and dihexyl esters, compounds with tetramethylnonylamines and C ₁₁₋₁₄ -alkylamines (CAS Reg. No. 80939-62-4).	For use only at levels not to exceed 0.5 percent by weight of the finished surface lubricant formulation.
Polyethylene glycol (molecular weight 300 or greater)	Mono- and diethylene glycol content not to exceed a total of 0.2 pct.
Stannous stearate.	
Stearic acid amide.	
Stearyl stearate.	
Tetrakis[methylene (3,5-di- <i>tert</i> -butyl-4-hydroxyhydrocinnamate)] methane (CAS Registry No. 6683-19-8).	For use at a level not to exceed 0.5 percent by weight of the finished surface lubricant formulation.
Triethylene glycol	Diethylene glycol content not to exceed 0.1 pct.
Wax, petroleum	Complying with § 178.3710.

(c) The substances identified in paragraph (a)(2) of this section may be used in surface lubricants used to facilitate the drawing, stamping, and forming of metallic articles from rolled foil and sheet stock provided that total residual lubricant remaining on the metallic article in the form in which it contacts food does not exceed 0.015 milligram per square inch of food-contact surface.

(d) Subject to any prescribed limitations, the quantity of surface lubricant used in the manufacture of metallic articles shall not exceed the least amount reasonably required to accomplish the intended technical effect and shall not be intended to nor, in fact, accomplish any technical effect in the food itself.

(e) The use of the surface lubricants in the manufacture of any article that is the subject of a regulation in parts 174, 175, 176, 177, 178 and §179.45 of this chapter must comply with any specifications prescribed by such regulation for the finished form of the article.

(f) Any substance that is listed in this section and the subject of a regulation in parts 174, 175, 176, 177, 178 and §179.45 of this chapter shall comply

with any applicable specifications prescribed by such regulation.

[42 FR 14609, Mar. 15, 1977, as amended at 48 FR 238, Jan. 4, 1983; 49 FR 10113, Mar. 19, 1984; 49 FR 29579, July 23, 1984; 50 FR 36874, Sept. 10, 1985; 52 FR 10223, Mar. 31, 1987; 54 FR 6124, Feb. 8, 1989; 54 FR 24899, June 12, 1989; 56 FR 55456, Oct. 28, 1991; 57 FR 23953, June 5, 1992; 58 FR 17513, Apr. 5, 1993; 64 FR 47110, Aug. 30, 1999; 69 FR 24512, May 4, 2004]

§ 178.3930 Terpene resins.

The terpene resins identified in paragraph (a) of this section may be safely used as components of polypropylene film intended for use in contact with food, and the terpene resins identified in paragraph (b) of this section may be safely used as components of polyolefin film intended for use in contact with food;

(a) Terpene resins consisting of the hydrogenated polymers of terpene hydrocarbons obtainable from sulfate turpentine and meeting the following specifications: Drop-softening point of 118°-138 °C; iodine value less than 20.

(b) Terpene resins consisting of polymers of beta-pinene and meeting the following specifications: Acid value less than 1; saponification number less than 1; color less than 4 on the Gardner